# Gas Chromatography-Mass Spectrometry

*Gas chromatography-mass spectrometry (GC-MS) is the most commonly used technique for high sensitivity analysis of complex mixtures of volatile and semivolatile organic compounds. This is one of the only techniques for organic analysis that provides unambiguous identification of analytes. The technique involves the chromatographic separation of individual components of a mixture of organic compounds and the production of a mass spectrum for each component. The mass spectrum is a unique fingerprint for each organic compound that enables positive identification of each sample component. This technique is qualitative and quantitative. Other sample introduction methods besides GC are available.*

## Principle of Technique

The sample is introduced onto a capillary chromatographic column via a heated injection port, and then separated by the partitioning between a mobile gas phase and a stationary phase in the column (see GC). The components travel to the mass spectrometer where they may be ionized by electron impact or chemical ionization processes. Chemical ionization mass spectra produce predominantly molecular ions, with few fragment ions. Electron impact ionization produces many fragment ions, which are useful for structure identification. The fragment ions are separated according to their mass-to-charge ratios by a quadrupole mass filter and detected and amplified by an electron multiplier. The result is a total ion chromatogram depicting time on the *x* axis and total ion abundance on the *y* axis. Additionally, each peak in the chromatogram has a unique mass spectrum, which can be used for compound identification.

## Purge and Trap

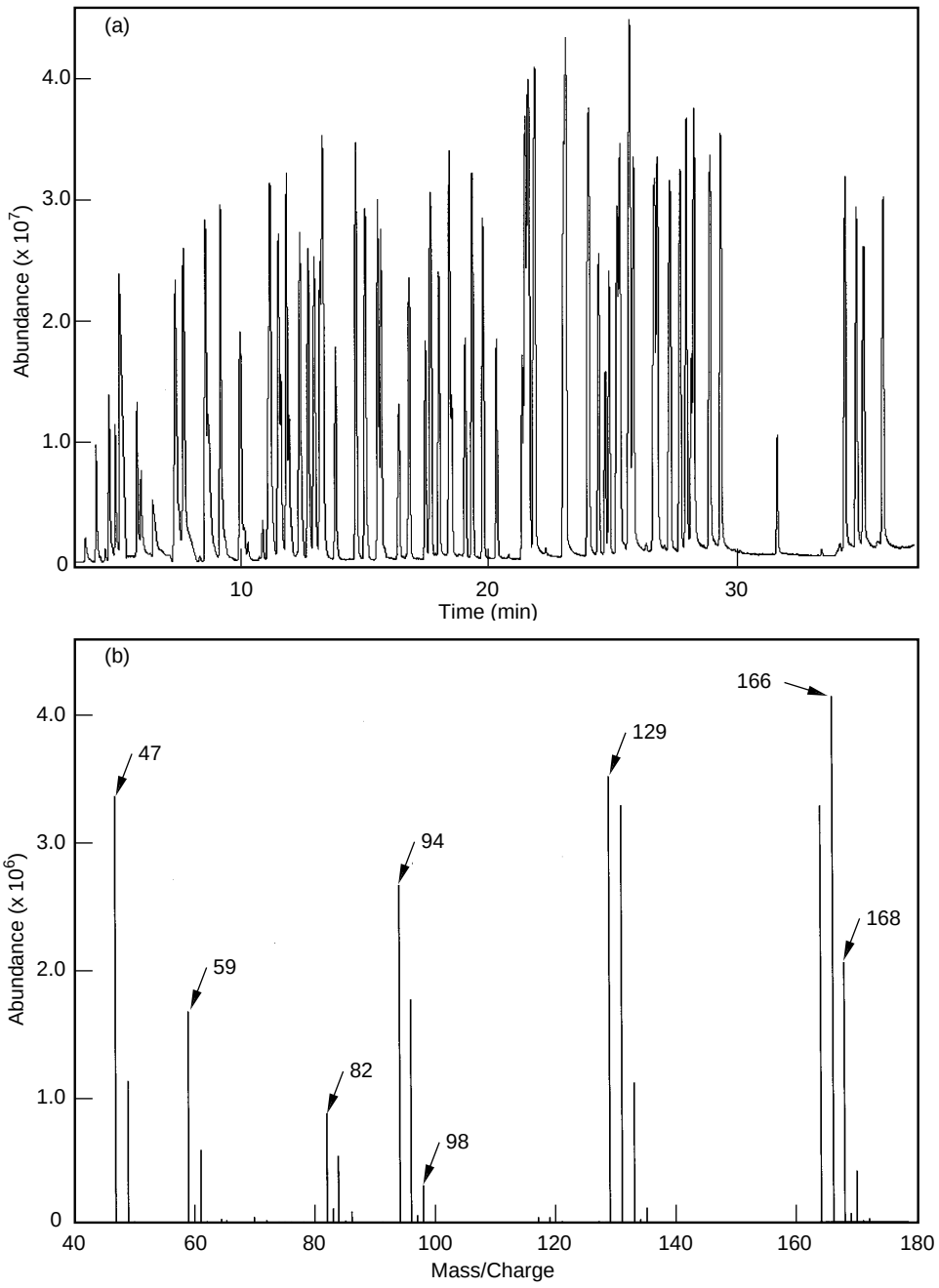
Several inlet configurations are available for GC-MS analysis. Purge and trap introduction involves bubbling helium carrier gas through an aqueous sample. Volatile compounds are expelled from the aqueous sample and trapped on a porous bed of charcoal, Tenax, OV-1, or other suitable sorbent held at room temperature. The trap is then flash heated to desorb the volatile compounds and carry them to the injection port of the GC for GC-MS analysis.

## Pyrolysis GC-MS

In pyrolysis GC-MS, a solid sample is placed in a quartz tube, which is then inserted into a platinum coil. The entire sample assembly is placed in a heated assembly attached to the front of the GC-MS system and swept with helium carrier gas. When the coil is rapidly heated (0.1 to 20°C/ms and at least 75°C/ms for ballistic heating) to 500–1000°C, pyrolysis of the sample material occurs. The pyrolysis products are carried to the injection port of the GC for GC-MS analysis. This technique is useful for fingerprinting of polymers. The pyrolysis probe can also be inserted directly into the mass spectrometer source.

## Examples of Applications

- Identification of trace contaminants in liquid and gas samples (pollutants in air, waste water, and solid waste).
- Identification of oils.
- Identification of additives, such as antioxidants and plasticizers.
- Identification of pure solids by direct inlet probe.
- Identification of drugs and metabolites.




GC-MS is used to perform EPA Method 8260 analyses of volatile organic compounds in environmental samples. In this example, the mass spectrum of the compound eluting from the GC column at 15.5 min can be identified as tetrafluoroethylene by its mass spectrum.

## Direct Insertion Probe

In direct insertion probe (DIP) MS, a solid sample is also placed in a quartz tube at the end of a probe, and inserted directly into the ion source of the mass spectrometer. Heating rates with a maximum of 200°C/min programmable (300°C ballistic heating) and final temperatures up to 400°C are much lower than for pyrolysis GC-MS. DIP-MS may be useful for the analysis of nonvolatile compounds and explosives.

## Samples

***Form.*** Solids, liquids, and gases; organic and some inorganic compounds.

***Size.*** Sample size must be sufficient to yield a few nanograms to micrograms of analyte. In special applications, picogram amounts of analyte may be detected.

Liquid samples should range from 1 to 3 μL, and contain a minimum of 10 ng of the compound of interest. For gases, about 10 ng of the analyte are needed. Depending on the concentration of the gas, injection volumes of 0.1 to 10 mL may be required. Samples of pure solids may be 1 mg or less.

***Preparation.*** The degree of sample preparation required depends on the sample matrix and the desired limits of detection for the analytes.

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## Limitations

GC-MS is applicable to liquids and gases—and to solids of sufficient volatility and thermal stability that can be dissolved in a suitable solvent. Compounds must be ionizable in the mass spectrometer.

The detection limit is often 1 ng (full scan) and is typically 20 ng, depending on how well the compound ionizes in the mass spectrometer. Picogram detection limits may be achieved for specific applications.

Strongly acidic or basic solutions will destroy the capillary columns used for separations.

## Estimated Analysis Time

At least 1 h is required for the GC-MS analysis of liquid or gas samples that contain one or two components of approximately known concentration (and which are suitable for the capillary column currently installed in the system). More complex mixtures require a minimum of 4 h. Instrument setup, calibration, and sample workup can increase the total analysis time by several hours or even days. Samples with unknown analyte concentrations usually require multiple analyses. Data analysis and interpretation can take from 15 min to several days. In general, the complexity of the analysis has the greatest effect on analysis time.

## Capabilities of Related Techniques

GC–Fourier transform infrared can perform functional group analysis and isomer identification but is at least an order of magnitude less sensitive than GC-MS.

GC with other detectors, liquid chromatography, and spectroscopic methods may also aid in the identification of unknowns and mixtures but these methods are less sensitive than GC-MS.

GC-MS-MS may provide additional information about compound structure and identification not available from GC-MS analysis.

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